Analysis of CO_2 Composite Spray Cleaning System Results

Nicole Chen
Jet Propulsion Laboratory, California Institute of Technology
4800 Oak Grove Dr.
Pasadena, CA 91109
818-354-4542
Nicole.C.Chen@jpl.nasa.gov

Ying Lin
Jet Propulsion Laboratory, California Institute of Technology
4800 Oak Grove Dr.
Pasadena, CA 91109
818-393-6381
Ying.Lin@jpl.nasa.gov

David Jackson
Clean Imagineering LLC
26074 Ave Hall Unit 6
Santa Clarita, Ca 91355
661-430-6768
David.jackson@cleanlogix.com

Shirley Chung
Jet Propulsion Laboratory, California Institute of Technology
4800 Oak Grove Dr.
Pasadena, CA 91109
818-354-4005
Shirley.Y.Chung@jpl.nasa.gov

***Abstract*—In a previous study, the cleaning efficiency of a CO_2 composite cleaning system for particulate removal was tested. The study covered particles from spores to fluorescent particles of different sizes as well as a variety of substrate surfaces, including aluminum, titanium, stainless steel, and nitinol. Particles were deposited using aerosol (dry) and droplet (wet) deposition. Results from the previous study show that the CO2 composite spray system is capable of cleaning to sterility for aerosol deposited spores and is capable of cleaning a minimum of a 4-log reduction for droplet deposited spores. This minimum 4-log reduction matches current Planetary Protection dry heat microbial reduction requirements. In this paper we will present new data to further correlate the cleaning efficiency with how contamination was introduced to the surface, the surface roughness, and particle size. Possible causes for such correlations will be discussed.**

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1. INTRODUCTION

In a previous study the cleaning efficiency of the Cleanlogix SnoPen, a CO_2 composite spray cleaning system, was evaluated. The study showed the CO_2 composite spray system is capable of cleaning to sterility for aerosol deposited spores and is capable of cleaning a minimum of a 4-log reduction for droplet deposited spores. This minimum 4-log reduction matches current Planetary Protection dry heat microbial reduction requirement. Since then, we have conducted additional experiments on spores deposited with 50% ethanol and reduced the particle size to 0.2µm.

During the previous study, contaminants in water were used for our deposition experiments. We found that particles pile up on edges of edges of the droplets as they dry, which created a non-uniform and non-monolayer contaminated surface (Figure 3).

The speculation was that multilayer deposits created an easier surface to clean. In this study we 50% ethanol/water suspension for droplet deposition which give a uniform deposited surface to further evaluate the CO_2 jet cleaning effectiveness.

We used fluorescent particles of 1um, 0.5um, and 0.2um with both dry deposition and wet deposition with 50% ethanol to evaluate the particle size effect on cleaning efficiency.

2. METHOD

Materials

The substrate material used in this study was nitinol with dimensions 2cm by 1cm by 0.2cm. The nitinol coupons had a surface roughness of 14 rms (root mean square microinches) and 3 rms. Nitinol rms 3 has a smoother surface than nitinol rms 14.

The contaminants used include Bacillus Atrophaeus bacterial spores, 1.0µm diameter FluoSpheres® Aldehyde-Sulfate Microspheres, 0.5µm diameter FluoSpheres® Carboxylate Modified Microspheres, and 0.2µm FluoSpheres® Sulfate Microspheres. The FluoSpheres were purchased from Life Technologies Corporation.

Sample Preparation

Sample substrates were cleaned prior to particle deposition. Coupons would be soaked in acetone for a minimum of 5 minutes in a fume hood. Following this, sample substrates would then be manually cleaned by acetone and isopropyl alcohol wiping. Then the substrates would be rinsed with

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acetone. Substrates that would undergo spore deposition were also UV sterilized on both sides for 15 minutes each. Once cleaned, the substrates were ready for particle deposition and were placed in sterile petri dishes and sealed with parafilm. Particles were deposited onto the cleaned substrates either through aerosol deposition (dry deposition) or droplet deposition (wet deposition).

With aerosol deposition, the contaminant particles were gently deposited onto the substrate surface. This was accomplished by using an aerosolizer shown in Figure 1. The goal was to deposit 10_5 particles onto the coupon surface. This deposition method simulates natural fallout onto the substrate surface.

Figure 1: Aerosolizer for aerosol deposition

Droplet deposition was accomplished using a pipette and contaminants were suspended in a dilution and deposited directly onto the substrate surface, as shown in Figure 2. Dilutions were vortex mixed and sonicated prior to deposition. A 10µL volume was then deposited directly onto the substrate using a pipette. The samples were then left to dry overnight in a Class 100 laminar flow hood. Spores from the previous study were deposited using pure water. This new study used spore and fluorescent particles suspended in a 50/50 ethanol/water mixture. This was done to ensure a more uniform distribution and drying of the fluorescent particles, as shown in Figure 4. Conversely, Figure 3 shows the non-uniform distribution and drying of the fluorescent particles when using a pure water mixture.

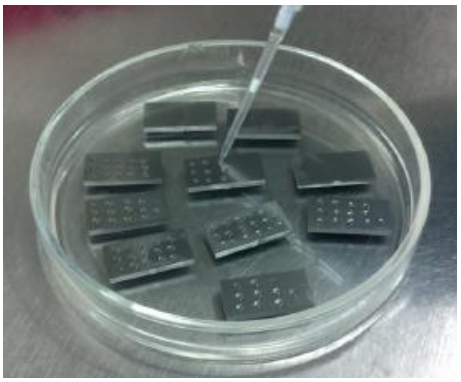

Figure 2: Droplet deposition on nitinol coupons

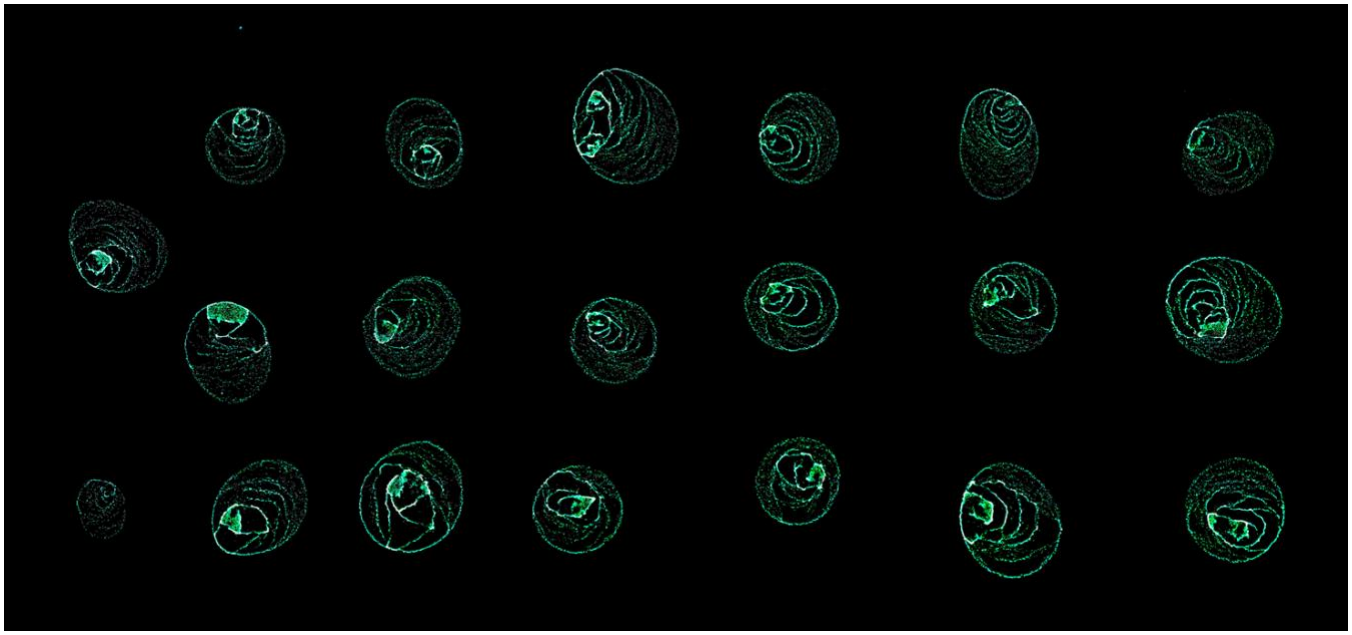

Figure 3: Image of fluorescent particles deposited using 100% water solution

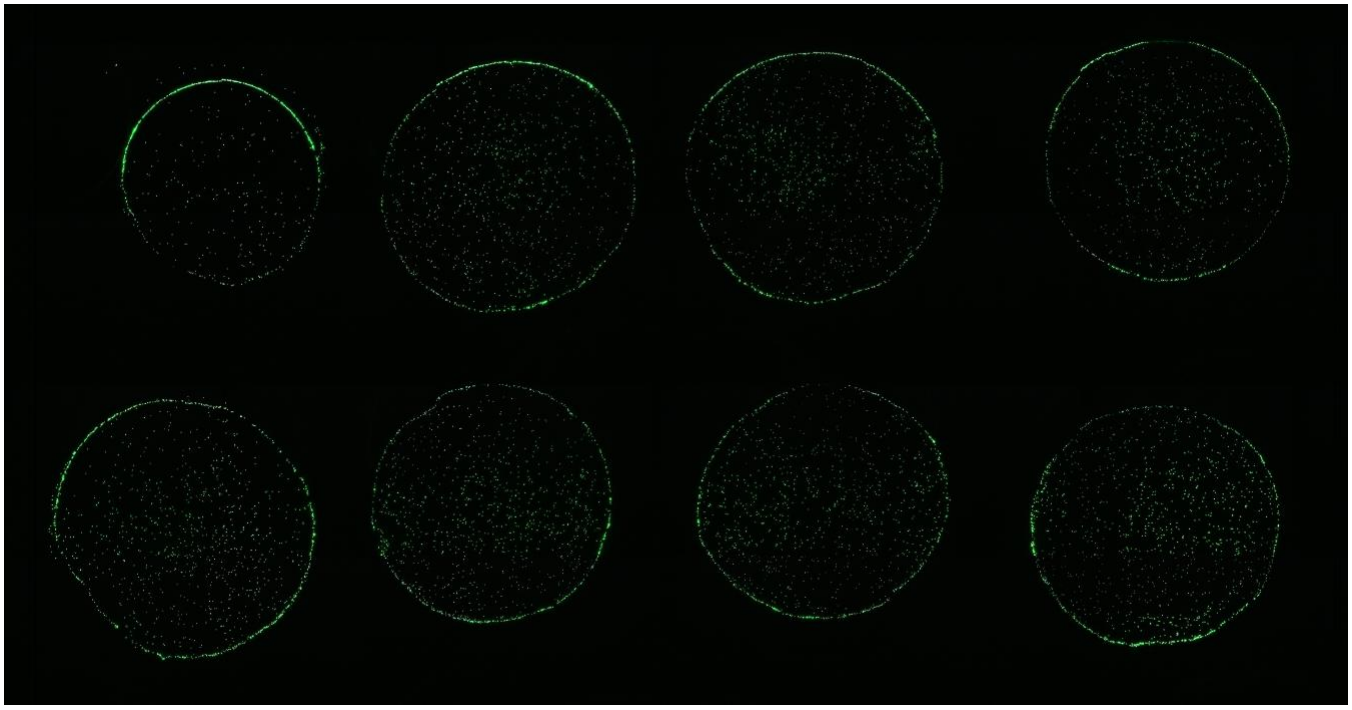

Figure 4: Image of fluorescent particles deposited using 50% ethanol/water solution

CO_2 Cleaning

After depositing particles onto the substrates, the samples are then cleaned using the Cleanlogix CO_2 SnoPen. The optimal parameter settings for spore removal were propellant (N_2) temperature 130°C, 40 psi propellant (N_2), CO_2 speed ~ 1.3 – 1.5 lbs. of CO_2 per hour, 45° incident to the sample surface, 0.5 inch distance from the sample surface, and 3 passes unidirectional passes across the length and width of the sample surface. All testing and cleaning were done in triplicate. Following CO_2 cleaning, samples were analyzed and a log reduction was determined based on before and after cleaning counts.

Spore Analysis

Spore samples were analyzed using the NASA standard assay procedures. After cleaning, the sample coupons were placed in individual sterile glass test tubes with 10mL of sterile water. The samples were then vortex mixed and sonicated to release remaining spores. Then 2mL of each sample tube was plated in tryptic soy agar (TSA), four plates per sample. The plated samples were then incubated at 32°C. Spore colonies, if any, were counted 24 and 48 hours after incubation.

If there are no spore colonies after 48 hours of incubation, then 10mL of sterile tryptic soy broth (TSB) is added to the test tube containing the cleaned sample. This is done to test for sterility. After 7 days of incubation at 32°C, if the coupon is sterile, then the broth will remain clear. However, if a single microbe is in the tube, regardless of origin, the sterility test will fail.

Fluorescent Particle Analysis

After cleaning, fluorescent particle samples were analyzed using a Zeiss fluorescent microscope. Sample substrates were placed onto the moving stage and a raster scan was performed across the sample surface. Images are taken of sections of the sample surface and stitched together to form a complete image of the entire surface. This image was acquired through the Axiovision program. The scanned image was then processed using ImageJ to count the particles.

3. RESULTS

The cleaning results are presented as a percent removal fraction in order to compare the results. The percentages are shown with 3 decimal places.

Table 1 shows the percent removal of spores (~ 1um in size) and 1µm fluorescent particles on nitinol using both aerosol and droplet deposition. Comparisons are drawn between some of the entries in Table 1 and shown in Figures 5 – 8.

Table 1: Spore and 1µm fluorescent particle cleaning data for aerosol and droplet deposited samples

Substrate	Contaminant	Deposition	Before (cfu)	After (cfu)	Percent Removal (%)
Nitinol rms 14	Spores	Aerosol	8.93E+05	0	100
			8.93E+05	0	100
			8.93E+05	0	100
Nitinol rms 14	Spores	Droplet (100% water)	2.32E+06	0	100
			2.32E+06	0	100
			2.32E+06	1	99.999
Nitinol rms 3	Spores	Droplet(100% water)	1.04E+06	1	99.99990
			1.04E+06	0	100
			1.04E+06	0	100
Nitinol rms 3	Spores	Droplet (50% ethanol)	5.58E+05	25	99.995
			5.58E+05	52	99.990
			5.58E+05	1	99.999
			5.58E+05	29	99.994
			5.58E+05	8	99.998
			5.58E+05	84	99.984
Nitinol rms 3	1µm fluorescent particle	Aerosol	9.54E+04	26	99.972
			6.90E+04	6	99.991
			7.79E+04	8	99.989
Nitinol rms 3	1µm fluorescent particle	Droplet (50% ethanol)	1.39E+03	196	85.899
			2.43E+03	261	89.259
			1.42E+03	150	89.436
			7.66E+03	32	99.582
			8.17E+03	4	99.951
			7.59E+03	50	99.341
			4.47E+04	132	99.704
			5.10E+04	691	98.645
			4.94E+04	193	99.609

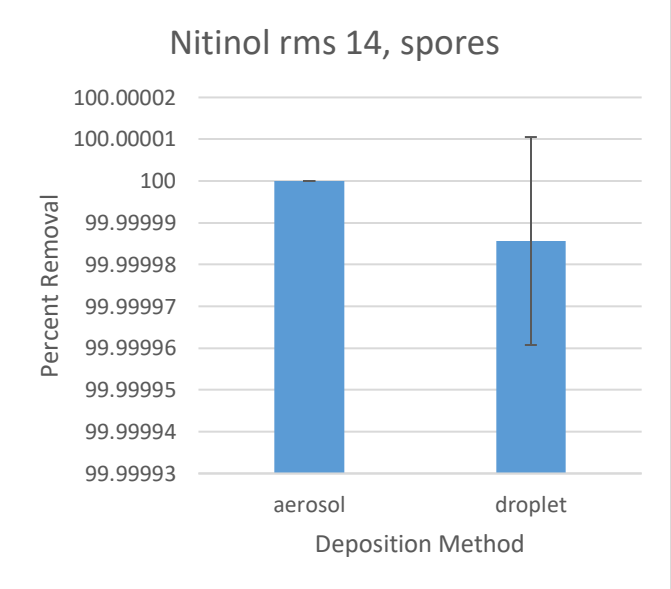

Figure 5: Cleaning efficiency for spore contaminants on nitinol rms 14 using aerosol and droplet deposition

Figure 5 shows a comparison of spore cleaning efficiency between aerosol (dry) and water droplet (wet) deposition on nitinol rms 14. Since both can be cleaned to a 99.999% removal, there is a negligible difference in cleaning effectiveness between dry and wet deposition of spores on nitinol rms 14.

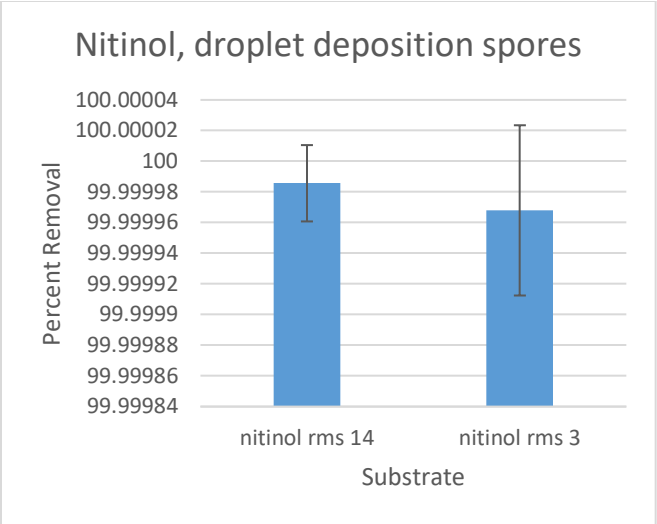

Figure 6: Cleaning efficiency for spore contaminants on nitinol with different surface roughness

Figure 6 shows a comparison of cleaning efficiency between substrates with different surface roughness values. Both can be cleaned to a 99.999% removal with a marginal difference. As shown in Table 1, there is no data for aerosol deposited spores on nitinol rms 3. The comparison shown in Figure 6 proves that cleaning nitinol rms 14 is comparable to cleaning nitinol rms 3. This means nitinol rms 14 and nitinol rms 3 can be compared interchangeably.

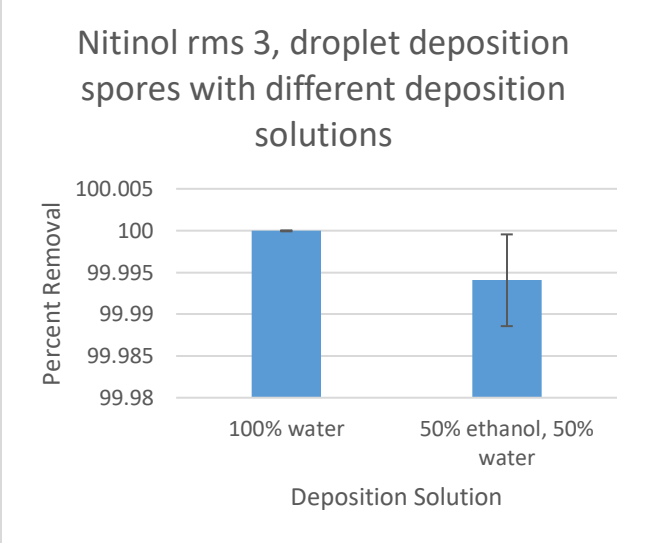

Figure 7: Cleaning efficiency for wet deposition of spore in different deposition solutions

Figure 7 shows a comparison of cleaning efficiency for droplet deposited spores using water and 50% ethanol, 50% water solution. The results show that the more uniform spore deposition with 50% ethanol make it harder to remove. However, even though the deposition with 50% ethanol solution has a slightly lower percent removal, it still can reach the 4 log reduction in cleaning.

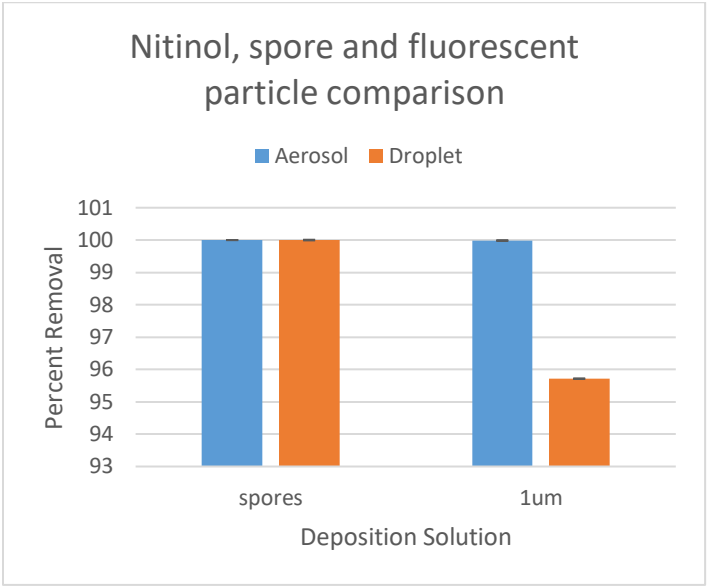

Figure 8: Cleaning efficiency on aerosol and droplet deposited spores vs. 1µm fluorescent particles

Figure 8 shows the comparison between spore contamination and 1µm fluorescent particle contamination using both aerosol and droplet deposition. 1µm fluorescent particles are used in this comparison because they are approximately the same size as spores. This ensures that size does not affect the results. The results show that the percent removal is comparable for both spores and 1µm fluorescent particles that have been deposited using aerosol deposition. However, there is a larger difference of 4.285% between the percent removal of droplet deposited spore contaminants and droplet deposited 1µm fluorescent particle contamination.

Table 2: Aerosol/Droplet deposited fluorescent particle cleaning data on nitinol rms 3

Substrate	Particle size	Deposition	Before (# of particles)	After (# of particles)	Percent Removal (%)
Nitinol rms 3	1.0µm	Aerosol	9.54E+04	26	99.972
			6.90E+04	6	99.991
			7.79E+04	8	99.989
Nitinol rms 3	1.0µm	Droplet (50% ethanol)	1.39E+03	196	85.899
			2.43E+03	261	89.259
			1.42E+03	150	89.436
			7.66E+03	32	99.582
			8.17E+03	4	99.951
			7.59E+03	50	99.341
			4.47E+04	132	99.704
			5.10E+04	691	98.645
			4.94E+04	193	99.609
Nitinol rms 3	0.5µm	Aerosol	1.33E+05	0	100
			1.30E+05	2	99.998
			1.21E+05	0	100
Nitinol rms 3	0.5µm	Droplet (50% ethanol)	1.03E+03	83	91.941
			1.30E+03	116	91.076
			9.30E+02	105	88.709
			7.68E+03	185	97.591
			7.56E+03	185	97.552
			7.22E+03	307	95.747
			3.09E+04	333	98.922
			4.48E+04	1411	96.850
			4.56E+04	1045	97.708
			1.49E+03	419	71.935
			6.55E+02	9	98.625
			7.85E+02	439	44.076
			6.22E+03	367	94.101
			1.48E+04	118	99.204
			4.76E+03	53	98.887
			7.59E+03	2851	62.427
			1.32E+04	3116	76.329
			1.05E+04	857	91.863
Nitinol rms 3	0.2µm	Aerosol	3.43E+05	20	99.994
			1.82E+05	11	99.993
			1.39E+05	5	99.996
Nitinol rms 3	0.2µm	Droplet (50% ethanol)	1.17E+03	328	71.869
			4.09E+02	380	7.090
			4.77E+02	385	19.287
			6.73E+03	2052	69.509
			2.28E+03	814	64.219
			2.71E+03	1218	55.088
			2.97E+03	1159	61.002
			1.67E+04	5423	67.598
			3.65E+03	2144	41.211

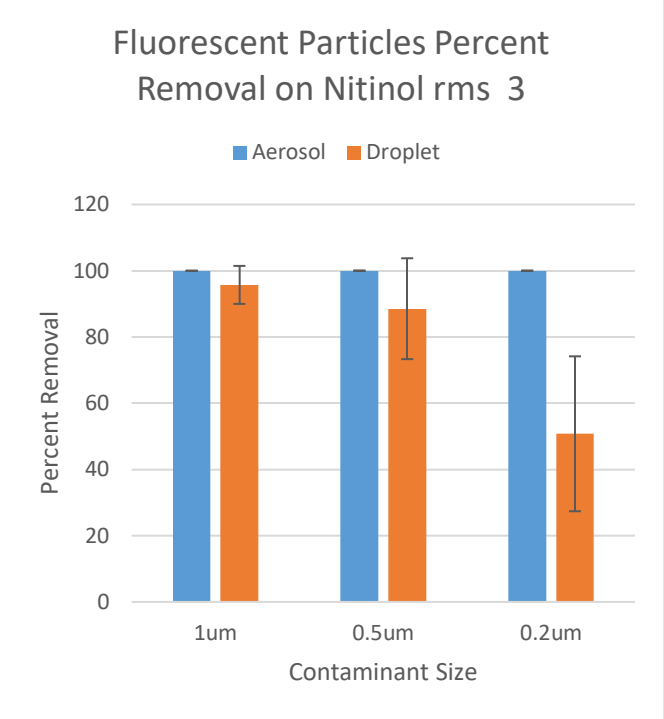

Figure 9: Fluorescent particle analysis, percent removal results summary for fluorescent particles of different sizes

Table 2 shows the cleaning data for aerosol and droplet deposited fluorescent particles of various size on nitinol rms 3. The results are summarized in Figure 9. Again, the effect of particle size on the cleaning efficiency is minimum with dry deposited contaminants. The effects are much more significant with the wet deposited contaminants. The smaller the particle, the harder it is to clean.

4. Discussion

As shown in Table 1 and 2, there is a difference in cleaning efficiencies between aerosol deposited spores and droplet deposited spores. In general, it is easier to remove spore or particle contamination that has been deposited using aerosol (dry) deposition versus droplet (wet) deposition in water. However, Figure 7 shows there is a slight difference in percent removal fraction between spores deposited using different droplet deposition solutions. This decrease in cleaning effectiveness is likely caused by the more even and distributed deposition caused by the addition of ethanol into the deposition solution. The ethanol in the deposition solution causes a uniform monolayer contaminated surface compared to the contaminated surface created when using a pure water deposition solution, as shown in Figures 3 and 4. It is much more difficult to remove this uniform monolayer deposition as the contaminant particles adhere directly to the surface and not on top of other contaminant particles.

Additionally, there is a difference in cleaning efficiencies between spores and fluorescent particles. Spores are approximately 1µm in diameter and are therefore comparable in size with the 1µm fluorescent particles. The comparison of these two is shown in Figure 8. The results show that there is a difference only when comparing spores with 1µm fluorescent particles that have been deposited using wet deposition. This implies that there is some sort of factor that affects the cleaning efficiency of droplet deposited fluorescent particles that does not affect spores. A likely explanation is that the intrinsic property or surface chemistry differences between spore contaminants and fluorescent particle contaminants causes this difference in cleaning effectiveness when using wet deposition.

One possible factor affecting adhesiveness is surface charge. The fluorescent particles used in this study are suspended in a mixture of water and a surfactant. The surfactant adds a surface charge to the fluorescent particles in suspension. This is done to prevent aggregation of particles. However, this added charge could be adding to the adhesiveness of fluorescent particles to the surface of our metallic coupon samples.

Finally, Figure 9 shows the comparison of percent removal for both aerosol and droplet deposited fluorescent particles of different sizes. There is a negligible difference in percent removal of aerosol deposited particles regardless of size. However, there is a marked difference between percent removal, and thus cleaning effectiveness, of droplet deposited particles. That is, it is much easier to remove larger particles that have been deposited using droplet deposition than it is to remove smaller particles that have been deposited using droplet deposition.

5. Conclusion

From the data shown in the above figures and tables, several comparisons were made. It is clear that spore contamination has a higher percent removal and thus a higher cleaning effectiveness from either dry or wet deposition. The new results demonstrated again, that CO2 jet cleaning is an effective technology to achieve greater than 4 log microbial reduction. It can be an effective tool to achieve planetary requirements. This is true despite differences in deposition method, surface roughness, and deposition solution. Additionally, the cleaning effectiveness of aerosol deposited spore contamination is comparable to aerosol deposited 1µm fluorescent particle contamination. There is a few percent difference of cleaning efficiency between droplet deposited spore contamination and droplet deposited 1µm fluorescent particle contamination. This is possibly due to property differences between spores and fluorescent particles such as surface charge and presence or absence of surfactants.

In general, aerosol deposited fluorescent particles of all sizes have a high removal percentage whereas droplet deposited fluorescent particles have a much lower percent removal. The size effect is only significant for the wet deposition samples.

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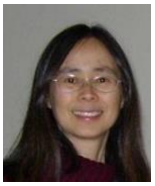
BIOGRAPHY

Nicole Chen *is a contamination control engineer at the Jet Propulsion Laboratory (JPL), California Institute of Technology. She received her BA in Physics from Occidental college. She has been involved with the development of the CO_2 composite spray cleaning system and has helped to optimize the system. Nicole is currently supporting the Mars 2020 mission and is the head contamination control engineer for the NISAR mission.*

Dr. Ying Lin *is a senior member technical staff at JPL’s biotechnology and planetary protection group. She has a BS in chemical engineering from Tsinghua University and a Ph.D. in chemistry from University of Arizona. She has extensive research experience in chemistry, biochemistry, and chemical engineering. She is the PI and Co-I of several proposals funded by JPL and NASA on planetary protection. She is currently a technical lead for the planetary protection tasks for Mars Technology Program.*



Shirley Y. Chung *is a contamination control engineer at the Jet Propulsion Laboratory, California Institute of Technology. She supported the Mars Science Laboratory project focusing on the Sample Acquisition / Sample Processing and Handling (SA/SPaH) system. Shirley also have extensive planetary protection implementation experience in various Mars flight projects including the Mars Exploration Rovers, Mars Reconnaissance Orbiter, Mars’01, Mars Microprobe DS2, MSP’98 Lander & Payloads, and Mars PathFinder. In addition to flight project work, she was involved in various R&D tasks such as using vapor hydrogen peroxide (VHP) as an alternative method to “sterilize” spacecraft hardware, materials compatibility study with VHP, biological cleaning efficiency studies, and organics cleaning study for the Mars Science Laboratory project.*

David Jackson *is President of CleanLogix LLC, a CO_2 technology development and licensing company, and Clean Imagineering LLC which provides clean manufacturing consulting services with unique expertise and technology for transforming precision cleaning, bonding, thermal coating, hard machining, and many other production processes. David began his career as group head of precision cleaning and contamination control at Hughes Aircraft Company in 1984, where he developed and patented first-generation CO_2 cleaning technologies. He holds a Bachelor's degree in Chemistry, has been issued more than 30 patents, and has commercialized multiple cleaning products utilizing CO_2 chemistry.*